Few-Layer Transition Metal Dichalcogenide Exfoliation, Characterization, and Modeling for Future Electronics

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Goal

- Exfoliate monolayer flakes of transition metal dichalcogenides (TMDs) for characterization and devices, modeling of electrical and mechanical device behavior

Motivation

- Thin TMD flakes exhibit unique electrical properties (high mobility, small band gap[1]) and mechanical properties (high flexibility[2])
- Attractive for construction of low-power, nanoscale devices and memory, leading to demand for reliable source of flakes for prototyping

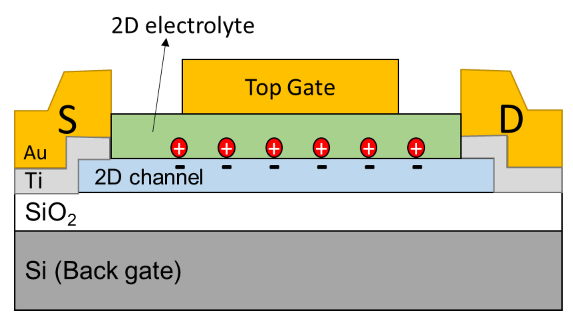

Fig. 1 Schematic of proposed device, using exfoliated MoS_2 and graphene as the 2D channel and top gate, respectively.

Exfoliation

- Mechanical exfoliation of thin flakes using the standard method, Figure 2.

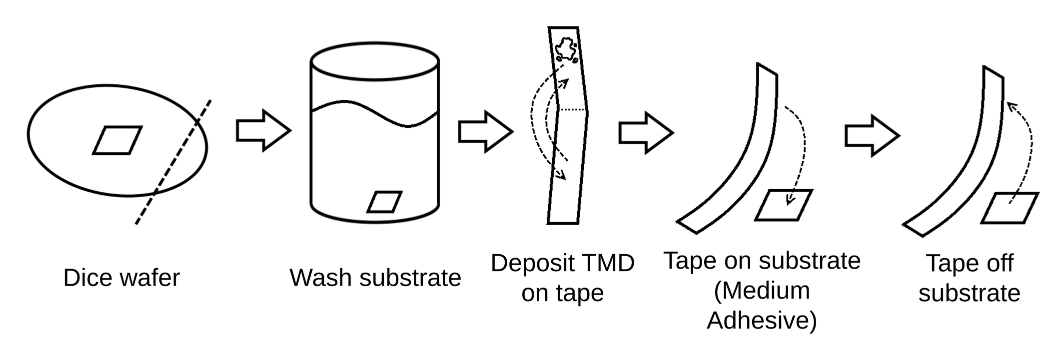

Fig. 2 Standard method for exfoliating thin flakes.

- Standard method gives candidate flakes for graphene, but for MoS_2 yields poor results (2-3μm lateral size, 10-15nm thick)
- A candidate flake for a device is longer than 15μm and thinner than 10nm
- Adding steps to standard method to increase yield of candidate flakes:
  - Thermal Annealing Pretreat[3]
    - Anneal substrate for 5 minutes at 100° C prior to exfoliation
  - Reactive Ion Etching (RIE) Pretreat[3]
    - Clean substrate with O_2 plasma prior to exfoliation
  - Varying levels of adhesive to perform exfoliation
    - Low (1009R-6.0), medium (18074-.50), high (Scotch)

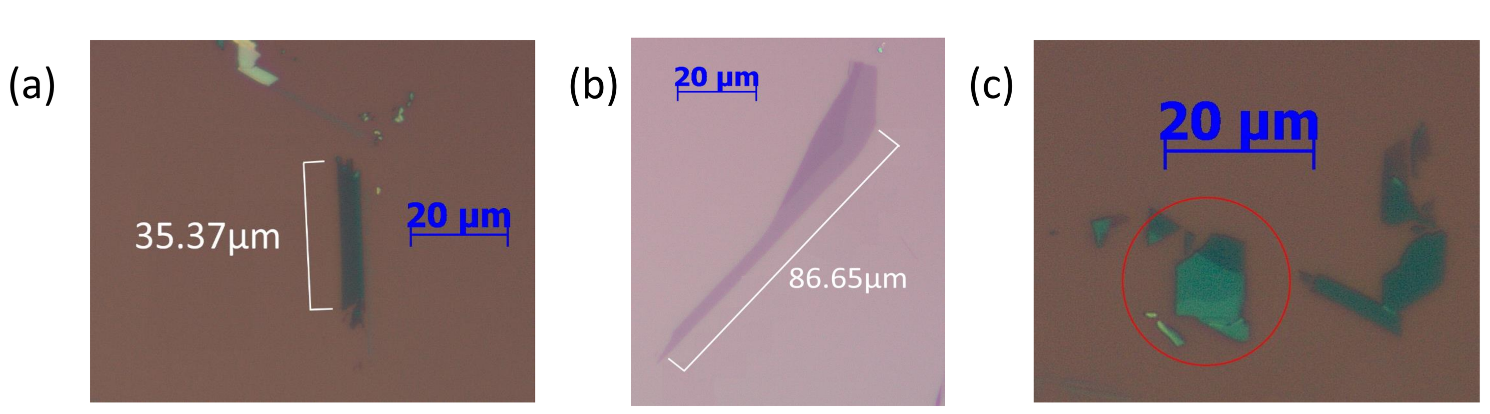

Fig. 3 Optical images of few-layer flakes. (a) Candidate MoS_2 flake exfoliated after thermal annealing pretreat. (b) Candidate graphene flake exfoliated using standard method. (c) An unusable flake (too thick).

Modified MoS_2 Exfoliation Method Results

- Annealing with the low adhesive tape (Fig. 4b) and annealing + RIE with medium adhesive tape (Fig 4a) gave the highest yield of candidate flakes, both with 1-2 tape depositions
  - RIE is an expensive and time-consuming process
  - Annealing with 1-2 tape depositions using low tape best option

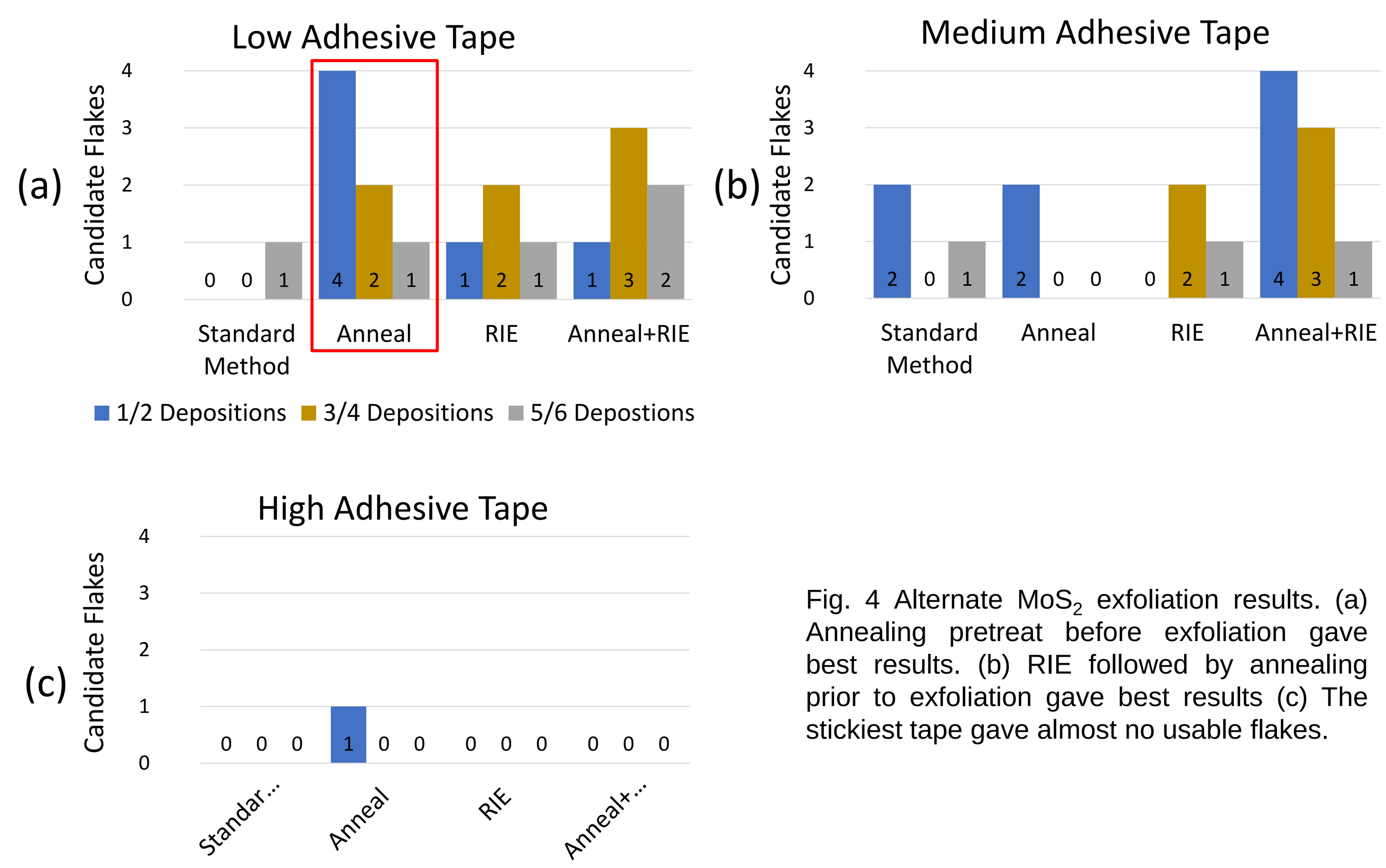

Fig. 4 Alternate MoS_2 exfoliation results. (a) Annealing pretreat before exfoliation gave best results. (b) RIE followed by annealing prior to exfoliation gave best results (c) The stickiest tape gave almost no usable flakes.

Modeling

- When some few-layer TMDs are bent, they undergo a phase transition from semiconductor to metal[4]
  - This phase transition happens at the least amount of strain (3%) in Molybdenum ditelluride ($MoTe_2$)[4]
  - Calculated to be reached at 3V with a channel length of 2.5μm
- To bend the flake, use an ionic polymer metal composite (IPMC)
  - In an IPMC, the anions are held in place, but the cations are free to move under bias. This causes a mechanical deformation of the material due to electrostatic pressure[5]
  - The strain ε on the plane can be found using the following equations[6], where α is the magnitude of steric strain, β is the electrostatic pressure, and γ is the crowding factor.
- Modeling in COMSOL Multiphysics by linking Transport of Dilute Species, Electrostatics, and Solid Mechanics modules.

$$\varepsilon = \frac{S-L}{L},\quad S = \frac{\theta}{K},\quad \theta = cos^{-1}\left(\frac{2R^2-L^2}{2R^2}\right),\quad K(V) = \alpha\sqrt{1-\gamma}\,\frac{V}{\sqrt{1+\frac{V}{8}}} + \frac{2}{3}\beta(1-2\gamma)\sqrt{2(1-\gamma)}V^{\frac{3}{2}}$$

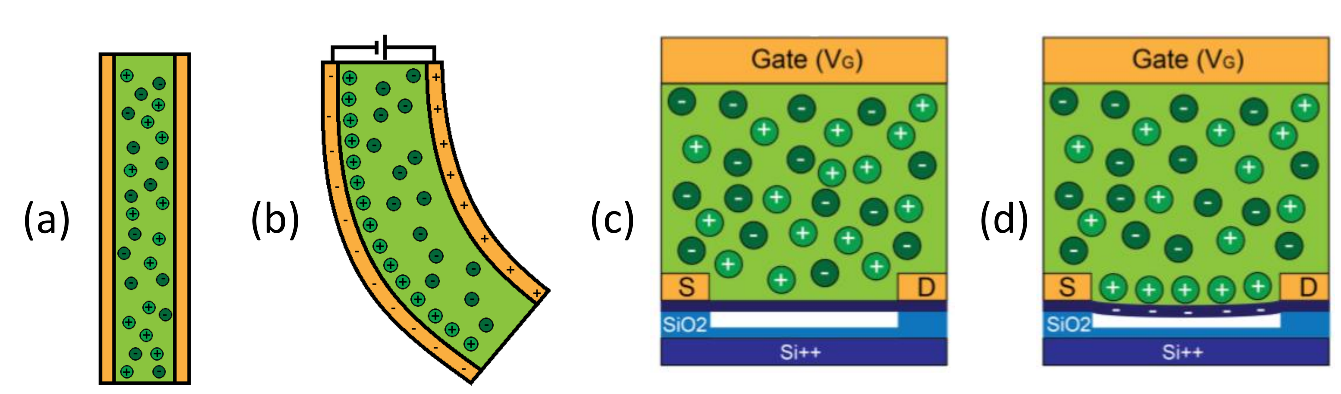

Fig. 5 (a) Schematic of IPMC device with no bias between the contacts. (b) Schematic of the IPMC device with voltage applied. (c) Device with no bias. (d) Device with V_G > 0, causing strain on $MoTe_2$

Characterization

- After exfoliation the lateral size, thickness, and roughness of the flake are measured using AFM
  - If the flake is too small (< 10μm length) it cannot be used in devices
- Roughness on MoS_2 flake using standard method is 0.425nm
  - Fig. 6a proves no residue left from annealing pretreat (R_Q = 0.42nm)
- Fig. 6b proves monolayer 2D electrolyte can be deposited homogeneously
- Step edge scan shows that MoS_2 flake in Fig. 4a is about 6 layers thick
  - Monolayer MoS_2 ~0.3nm thick
- Raised bumps in Fig. 6d caused by dirty substrate
- Wrinkle in Fig. 6f caused by topmost MoS_2 layers shifting

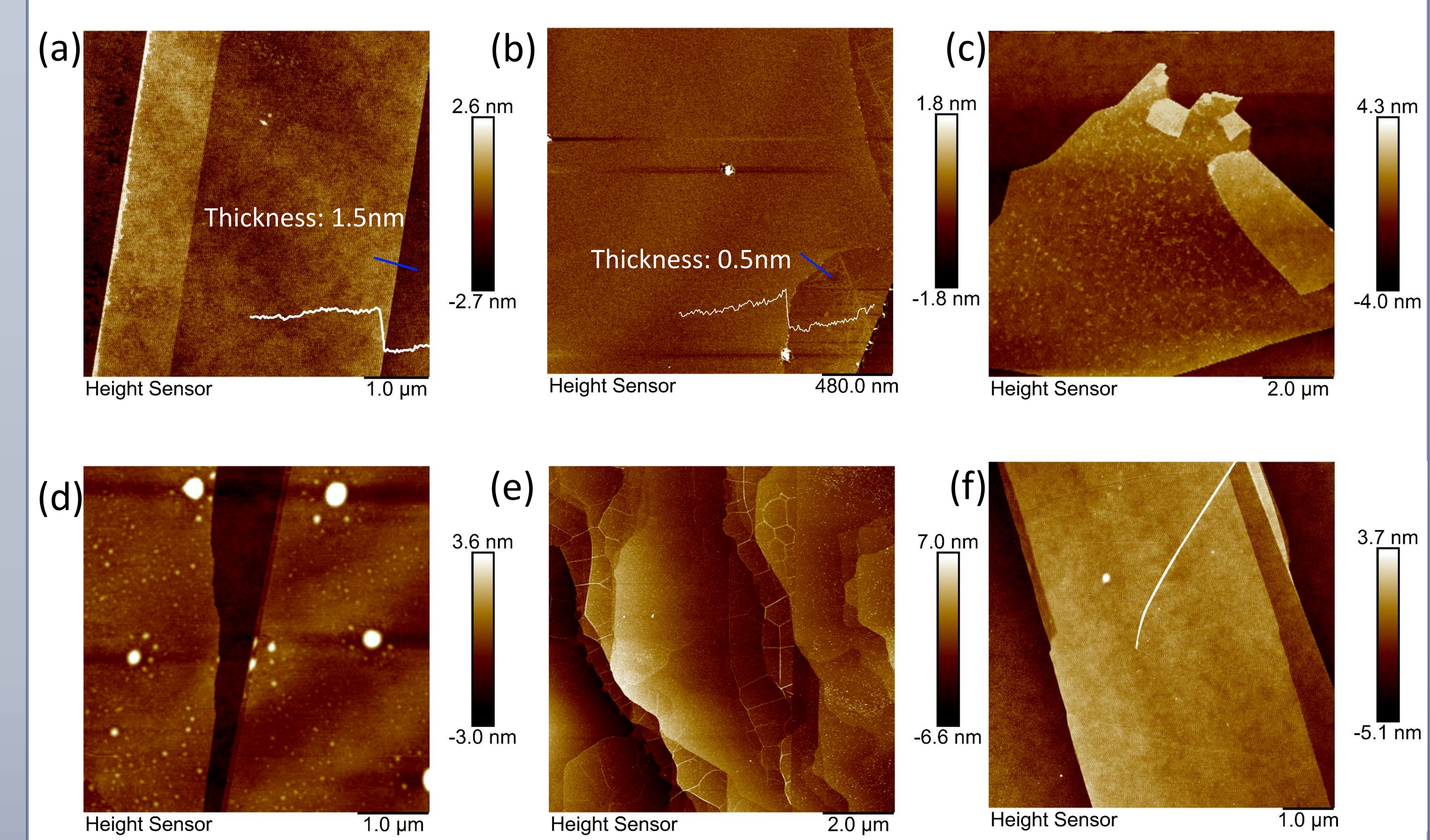

Fig. 6 AFM images of few-layer flakes. (a) Step edge of uncoated MoS_2 flake, exfoliated after annealing pretreat. (b) Step edge on homogeneous CoCrPc monolayer on exfoliated graphene. (c) Bare boron nitride (BN) flake, used as an insulator. (d) A close-up of a crack in a MoS_2 flake, exfoliated without annealing step. (e) Epitaxially grown graphene channel after photoresist removal. (f) Wrinkled MoS_2 flake.

Conclusions and Future Work

1. Adding the thermal annealing step during exfoliation allows for more reliable flake thickness and lateral size in MoS_2
2. The difference in exfoliation results between graphene and MoS_2 suggests that each material may need a different process or substrate.
3. Annealing does not leave any sticky residue from the tape on the flakes.

Future work:

- Explore the use of other TMD materials such as Tungsten diselenide (WSe_2), and exfoliation methods
- Use C-AFM for measuring electrical properties of flakes/coatings
- Use COMSOL to determine electrical and mechanical properties of devices specific to $MoTe_2$ and our polymer.

References
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